



Appl. No 09/991,702  
April 20, 2005

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicant: Gino Palumbo  
Appl. No: 09/991,702  
Filed: November 26, 2001  
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Examiner: S. Ip

Confirmation No: 3992

Docket No: AUST3001/JDB  
Customer No: 23364

**DECLARATION UNDER 37 C.F.R. 1.132**

Commissioner of Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

We, Karl T Aust and Klaus Tomantschger, hereby declare:

1. Karl T. Aust has a PhD in metallurgy from University of Toronto and between 1967 and 1991 was professor at the Department for Metallurgy & Material Science at the University of Toronto, and professor emeritus from 1991 to the present. He is presently a senior scientific advisor to Integran Technologies, the assignee of the instant patent application.
2. Klaus Tomantschger has a PhD in chemical engineering from the Technical University in Graz, Austria. He is presently vice president, intellectual property at Integran Technologies.
3. In the office action March 28, 2005 the examiner takes the position (page 3, page 4) that applicant "acknowledged prior art admission in page 4, first paragraph of instant specification discloses the features including the claimed special grain boundaries up to 29%".

The definition of special grain boundaries as lying within the specific coincident site lattice misorientations having  $\Sigma \leq 29$ , which are highly resistant to intergranular corrosion, and the percentage of special grain boundaries  $f_{sp}$  are clearly defined in the specification (page 4, lines 6-17 and page 13, lines 11-22) and it appears that the examiner is confusing the reciprocal density of common

Appl. No 09/991,702  
April 20, 2005

lattices points  $\Sigma$  with the fraction of special grain boundaries  $f_{sp}$ , expressed as the percentage fraction of special grain boundaries lying within  $\Sigma \leq 29$  to the total grain boundaries lying within  $\Sigma \leq 29$  and  $\Sigma > 29$ .

The  $\Sigma$  value; which is the inverse fraction of coincident sites (see Kronberg, and Wilson. Trans. Met. Soc. AIME, 185, 501 (1949) Brandon, Acta Metall., 14, 1479 (1966), is essentially a measure of the degree of order or periodicity in a grain boundary structure; lower values of  $\Sigma$  corresponding to more ordered structures. "Special" grain boundaries are described by the  $\Sigma$  relationship:  $\Sigma \leq 29$  with  $\Delta\theta \leq 15^\circ \Sigma^{-1/2}$  (see equation 1, page 13), where  $\Delta\theta$  is the maximum angular deviation from the specific  $\Sigma$  value.

Grain boundaries characterized as low  $\Sigma$  (high periodicity or well-ordered) boundaries, generally  $\Sigma \leq 29$ , are found to display improved physical and chemical properties as compared to general or high  $\Sigma$  (low periodicity) boundaries, generally  $\Sigma > 29$ .

The objective of the grain boundary engineering is to deliberately increase the frequency, i.e., the number fraction  $f_{sp}$  of low  $\Sigma$  boundaries ( $\Sigma \leq 29$ ) in conventional polycrystalline materials in order to improve the bulk material properties beyond previous limitations.

Page 4, lines 6-17 and page 13, lines 11-22 of the application referred to above, are set forth below with the underlined phrases being added

Page 4, lines 6-17:

Various studies have shown that certain special grain boundaries, described on the basis of "Coincident Site Lattice" model of interface structure (Kronberg, and Wilson. Trans. Met. Soc. AIME, 185, 501 (1949)(copies were filed with the IDS), as lying within  $\Delta\theta$  of  $\Sigma$ , where  $\Sigma \leq 29$  and  $\Delta\theta \leq 15^\circ \Sigma^{-1/2}$  (Brandon, Acta Metall., 14, 1479 (1966) copies were filed with the IDS) are highly resistant to intergranular degradation processes such as corrosion, cracking, and grain boundary sliding; the latter being a principal contributor to creep deformation. However, these studies provide no instruction as to how to achieve a high concentration of special grain boundaries, and as noted, it is only recently that techniques such as Orientation Imaging Microscopy have become available to determine the concentration of special grain boundaries in a polycrystalline material. Moreover, the only means of creating new special grain boundaries during solid state processing is to effect recrystallization of a material by deformation followed by suitable heat treatment; such a novel approach to the processing of lead acid battery positive current collectors therefore forms the basis of the present invention.

Page 13, lines 11-22:

In the present invention, the lead alloy positive current collector components of the battery are provided with a metallurgical microstructure having a high percentage, that is over 20%, 30%, 40% or 50%, of special grain boundaries. Special grain boundaries can be defined crystallographically as lying within

$$\Delta\theta \leq 15^\circ \Sigma^{-1/2} \quad (1)$$

(D.G. Brandon: Acta. Metallurgica. Vol 14, Page 1479, 1966; copies were filed with the IDS)

of specific coincident site lattice misorientations having  $\Sigma \leq 29$ . In this specification, including the claims, the term 'special grain boundaries' defines grain boundaries having  $\Sigma \leq 29$  and complying with equation 1.

4. In the office action March 28, 2005 the examiner takes the position (page 6) that "objective 1 in declaration, samples #3a and #2b show grain boundaries less than 40% which is found inconsistent with the claimed 40% (claim 42) and at least 50% (43)". In the experiments of the declaration, Pb-0.065Ca-1.4Sn alloy was exposed to one processing cycle of deformation for comparison purposes because Yasuda in his only example used only one cycle of deformation.

The Pb-0.065Ca-1.4Sn alloy, when exposed to one processing cycle of deformation and 250C/10min heat treatment showed an increase in  $f_{sp}$ , #3a from  $f_{sp} < 10\%$  to  $f_{sp} = 35\%$ , coinciding with a drop in hardness from 12.2 to 8.3HV and #2b from  $f_{sp} < 10\%$  to  $f_{sp} = 37\%$ , coinciding with a drop in hardness from 12.2 to 7.5HV. As highlighted in the specification (page 18, lines 30-34 and page 19, lines 5-26), Pb alloys with 0.065% Ca fall into the Class II alloys where "two or more cycles of deformation ...and recrystallization, ....will yield a microstructure consisting of a special grain boundary content of greater than 50% (Page 19, lines 21-26).

The results of objective 1 in the declaration for a class II alloy exposed to a single deformation/annealing cycle are therefore clearly anticipated and expected based on the disclosure in the specification and, as outlined in the specification, additional deformation/annealing cycles would further raise the special grain boundary fraction ( $f_{sp}$ ) and produce a material with  $f_{sp} > 50\%$ , thereby further enhancing the corrosion performance of the processed material. (see e.g. example #6, page 25, 26 for Pb-0.07Ca-1.4Sn processed in three

Appl. No 09/991,702  
April 20, 2005

deformation/annealing cycles (page 25, lines 29-31) to  $f_{sp} = 68\%$  (Table 4, page 26).

5. That all statements made herein of our knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code; and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

April 20, 2005  
Date:

Karl T. Aust  
Prof. Dr. Karl T. Aust

April 20, 2005  
Date:

Klaus Tomantschger  
Dr. Klaus Tomantschger